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TESTING OF LITHIUM-SULFUR DIOXIDE CELLS FOR WASTE DISPOSAL HAZA--ETC(U)

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RESEARCH AND DEVELOPMENT TECHNICAL REPORT

DELET-TR-79-0285-F

TESTING OF LITHIUM-SULFUR DIOXIDE CELLS FOR WASTE DISPOSAL
HAZARDS

David B. Boies
WAPORA, INC.
6900 Wisconsin Avenue
Chevy Chase, MD 20015

OCTOBER 1980

Final Report for Period Sep 79 - Mar 80

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ELECTRONICS TECHNOLOGY & DEVICES LABORATORY

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Lithium-sulfur dioxide cells have been examined to determine if they are considered hazardous wastes under Section 250.13 of the proposed amendment of 40 CFR, Part 250, as published in the <u>Federal Register</u> , Vol. 43, No. 243, 18 December 1978. Fresh and discharged samples of cells, manufactured by both P. R. Mallory, Inc., and Power Conversion, Inc., have been tested. The cells are not hazardous for disposal if the case is not ruptured. On the basis of the Structural Integrity Procedure of the proposed rules, the cells will not (continued)		

20. (continued)

rupture, and thus will not be considered hazardous. If the cell case should be ruptured in some manner, a small amount (approximately 2 to 12 grams/cell) of sulfur dioxide will be released. This is an irritant, odorous, and toxic gas; the degree of hazard will depend on the number of cells ruptured at a time, meteorological conditions, the distance to sensitive receptors, etc.

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The information in this report is submitted according to the requirements of Section 250.15(c) of the proposed amendment of 40 CFR, Part 250, as published in the Federal Register, Vol. 43, No. 243, 18 December 1978.

A. GENERAL INFORMATION

1. Identification of party desiring the demonstration:

US Army Electronics Research and Development Command
Fort Monmouth, New Jersey 07703.
2. Name and address of facility performing the demonstration:

WAPORA, Inc.
6900 Wisconsin Avenue
Chevy Chase, Maryland 20015
David B. Boies, Senior Chemical Engineer
3. The following samples of the waste were supplied by E. H. Reiss, US Army Electronics R & D Command, Fort Monmouth, New Jersey

40 LO-26SX cells manufactured by P. R. Mallory, Inc.:
 20 cells fresh
 20 cells discharged to 2.0 volts or less.

4 BA-5590 batteries manufactured by Power Conversion, Inc., each battery containing 10 cells:
 2 batteries (20 cells) fresh
 2 batteries (20 cells) discharged to 20 volts per battery (an average of 2 volts per cell).
4. The testing of the waste was carried out by David B. Boies, Senior Chemical Engineer, WAPORA, Inc., Chevy Chase, Maryland; and Mark Brandl, Associate Chemist.
5. The samples were supplied during October 1979.
6. The testing of the samples took place during October 1979 to February 1980.
7. The quantity of wastes that will be generated will depend on battery use, and is not certain at this time.
8. The wastes being tested consist of "D"-size cells operating with lithium and sulfur dioxide as the active ingredients. The individual cells are 3.3 cm (1.3 in) in diameter, by 5.7 cm (2.25 in) long, and may be discarded as batteries containing 2, 3, 5, or 10 cells. The cells contain a lithium anode, a carbon/Teflon/aluminum cathode, and a sulfur dioxide/lithium bromide/acetonitrile electrolyte, in a steel case. The cells are under pressure due to

the presence of the liquid sulfur dioxide. Table 1 gives the composition of a typical cell, and Figure 1 gives the vapor pressure of sulfur dioxide as a function of temperature. The cells are equipped with a venting mechanism to release the pressure if it becomes too high; a typical cell will vent at about 400 psi, which is reached at about 100° to 105°C for a fresh cell and at higher temperatures for discharged cells.

Table 1. Typical Composition of a Lithium-Sulfur Dioxide "D" Cell.

<u>Component</u>	<u>Material</u>	<u>Weight (grams)</u>
Anode	Lithium	2.6 to 3.0
Cathode	Carbon/Teflon/aluminum	13
Separator	Polypropylene	1.0
Electrolyte	Sulfur dioxide	23.5 to 24.5
	Acetonitrile	7.7 to 8.1
	Lithium bromide	2.3 to 2.5
Container	Steel	

B. WASTE SAMPLING

1. Cell Type and Condition

Cells from two manufacturers, Power Conversion, Inc., (PCI) and P. R. Mallory Inc., were obtained and were tested in both the discharged and fresh conditions.

2. Preparation of Representative Samples

- a. Whole cell. An individual cell was treated as a representative sample.
- b. Internals. Representative samples of the cell internals were obtained by cooling an individual cell to -40°C (-40°F), and the top was removed by a shallow saw cut through the cell wall just below the top. Liquid, if present, was removed by decanting. The internals were removed. They consisted of three layers, anode/separator/cathode, spiral wound. These layers were separated and cut into smaller pieces. A representative sample consisted of amounts of the liquid, anode, separator, and cathode, proportional to their weights in the cell. The sample internals were stored in covered glass containers at -40°C until used for testing.

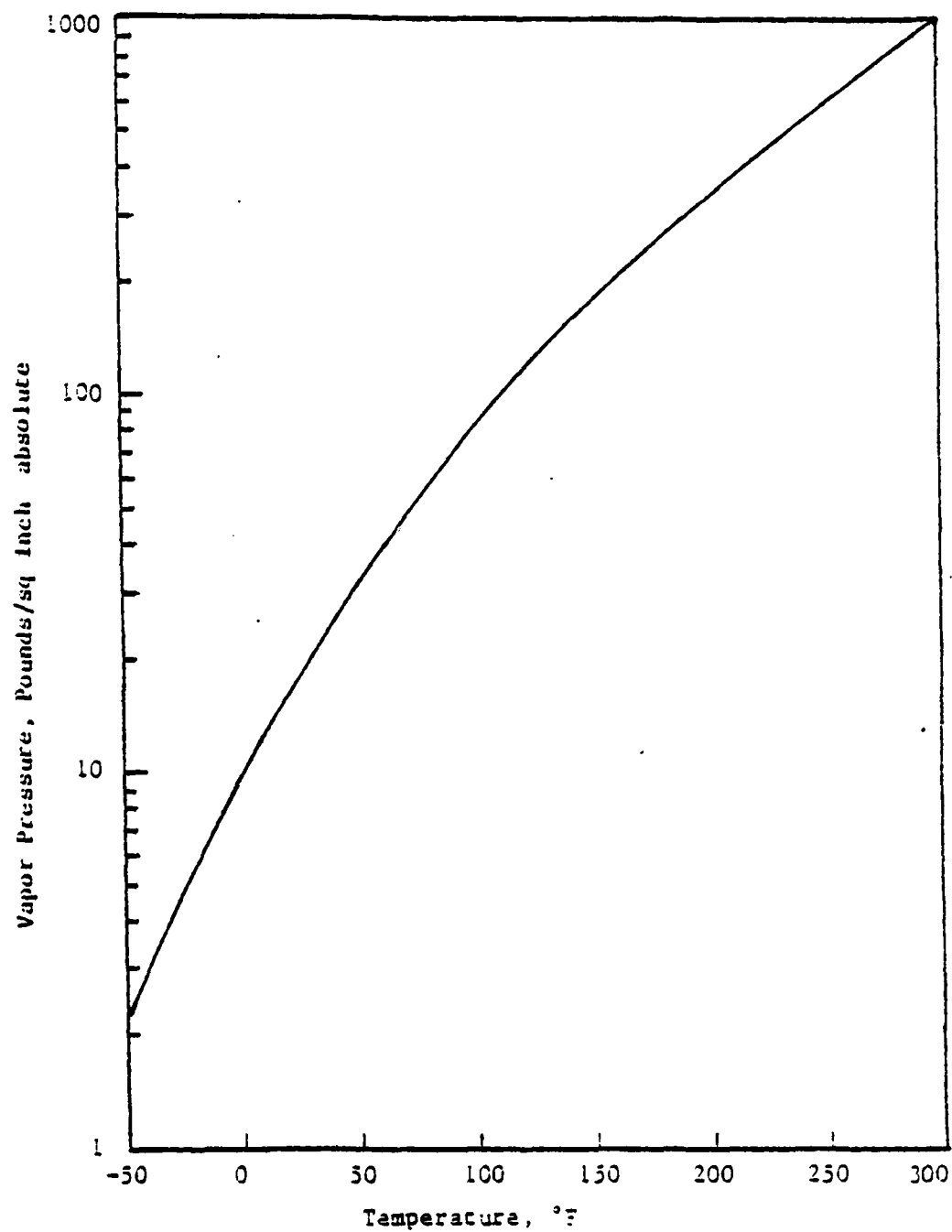


Figure 1. Sulfur Dioxide Vapor Pressure.

Source: Handbook of Chemistry and Physics. 59th Ed. Ed. by Robert C. Weast. 1978.

C. WASTE TESTING RESULTS

1. Individual Cell Tests

a. Test Procedures

It is the purpose of the proposed rules to test the waste for degree of hazard in the form in which it will be present in the disposal operation. Because the subject wastes will be disposed of in the form of batteries containing various numbers of cells, the tests should be conducted on the individual cells, the smallest common component. The Structural Integrity Procedure described in 250.13(d)(2)(B) of the proposed rules was applied to the individual cells. This consists of dropping a 330 g (0.73 lb) weight for a distance of 15 cm (6 in) onto the cell. This is repeated 15 times. It was found that in all cases the integrity of the individual cells was not changed, so that the individual cell could be considered a representative sample.

b. Test Results

The determination of the hazardousness of the waste based on the use of an individual cell as a representative sample is presented below according to the criteria presented in the various paragraphs of Section 250.13 of the proposed rules.

• (a) Ignitable waste:

(a)(1)(i): The criteria from this paragraph read as follows:

... is a liquid and has a flash point less than 60°C (140°F) determined by the method cited below or an equivalent method.

These criteria are not applicable, as the waste is not a liquid.

(a)(1)(ii): The criteria from this paragraph read as follows:

... is not a liquid and is liable to cause fires through friction, absorption of moisture, spontaneous chemical changes, or retained heat from manufacturing

or processing, or when ignited burns so vigorously and persistently as to create a hazard during its management.

The cells are in sealed steel containers and are not hazardous under the conditions listed in this paragraph.

(a)(1)(iii) and (iv): The criteria from these paragraphs read as follows:

. . . is an ignitable compressed gas as defined in 49 CFR 173.300(b), or is an oxidizer as defined in 49 CFR 173.151.

Although the waste contains a compressed gas, sulfur dioxide, it is not an ignitable compressed gas, nor is the waste an oxidizer.

• (b) Corrosive waste:

(b)(1)(i) and (ii): These criteria read as follows:

. . . is aqueous and has a pH less than or equal to 3 or greater than or equal to 12 as determined by the method cited below or an equivalent method, or corrodes steel (SEA 1020) at a rate greater than 0.250 inch per year at a test temperature of 130°F as determined by the method cited below or an equivalent method.

The wastes are sealed cells, and the criteria are not applicable.

• (c) Reactive waste:

(c)(1)(i): The criteria from this paragraph read as follows:

. . . is normally unstable and readily undergoes violent chemical change without detonating; reacts violently with water, forms potentially explosive mixtures with water, or generates toxic gases, vapors, or fumes when mixed with water; or is a cyanide or sulfide bearing waste which can generate toxic gases, vapors, or fumes when exposed to mild acidic or basic conditions.

The individual cells are sealed units and do not react in any of the ways cited in this criterion.

(c)(1)(ii): The criteria from this paragraph read as follows:

. . . is capable of detonation or explosive reaction but requires a strong initiating source or which must be heated under confinement before initiation can take place, or which reacts explosively with water.

The individual cells are sealed and do not react with water, but fresh cells do contain a considerable amount of stored energy that could be released rapidly by internal reaction as the temperature is raised, especially as the melting point of lithium is reached. To test this possibility, individual cells were placed in an oven at 204°C (400°F), which is above the melting point of lithium, 179°C (354°F). The cells vented sulfur dioxide, but there was no explosive reaction. It is probable that the venting of the oxidant, sulfur dioxide, took place before any violent internal reaction could be initiated.

(c)(1)(iii): The criteria from this paragraph read as follows:

... is readily capable of detonation or of explosive decomposition or reaction at normal temperatures and pressures.

As discussed previously, these cells, when fresh, contain a considerable amount of stored energy. To determine the effect of the rapid release of this energy, fresh cells were short-circuited by means of No. 12 copper wire. Although the temperature rose, the cells did not explode.

(c)(1)(iv): The criteria in this paragraph read as follows:

... is a forbidden explosive as defined in 49 CFR 173.51, a Class A explosive as defined in 49 CFR 173.53, or a Class B explosive as defined in 49 CFR 173.58.

The cells are not listed as forbidden explosives.

• (d) Toxic waste:

(d)(1): The criteria in this section deal with determination of the toxicity of a waste. The waste is prepared by subjecting it to a structural integrity procedure, leaching under defined conditions, and testing the leachate for the presence of certain listed contaminants. The individual cells, as discussed earlier, were not damaged by the structural integrity procedure, and as they are sealed cells in a steel case, nothing can be leached from the cells. Therefore, the individual cells are not toxic according to the criteria of this section.

2. Testing of Cells with Exposed Internals

The previous section of this report discussed the testing of individual cells for hazardous characteristics according to the EPA proposed rules. It was concluded that the cells would not be classified as hazardous as they are completely sealed, and their sealed nature is not destroyed by the Structural Integrity Test. It was also decided to test

the cells with the internals exposed to determine the hazards if by some means the structure of the cell was breached. This is most likely to happen if the cell is crushed by some force considerably greater than that employed in the Structural Integrity Test.

a. Test Procedures

Test methods, as listed in the proposed rules, Section 250.13, were used.

The following sections give some specific details of the methods used.

Section 250.13 is reproduced as an Appendix to this report.

- (a) Ignitable waste: The Setaflash Close Cup Tester was used.
- (b) Corrosive waste: The corrosion test procedure was based on the practices recommended in NACE Standard TM-01-69 (1976 Revision), published by the National Association of Corrosion Engineers. The controlling factor in the test procedure design was the fact that only a small amount of liquid (about 2 ml) was available from each cell that did not evaporate at the test temperature of 54°C (140°F).

Test specimen: Annealed mild steel wire, 1.6 mm (0.063 in) in diameter, by approximately 25 mm (1.0 in) long. The specimen was polished by aluminum oxide abrasive cloth graded from 50 to 120. After polishing, the wire was degreased by scrubbing with scouring powder and rinsing in water and acetone. The appropriate lengths were then cut, and the specimens coiled. Weight of the specimens was determined to 0.1 mg.

Test container: Pyrex test tube, 16 mm diameter, covered by polypropylene film. During the test, the tube was partially immersed in a water bath at 54°C (140°F).

Procedure: The liquid obtained from the Mallory and PCI fresh cells was allowed to warm to room temperature, the sulfur dioxide evaporating off. The discharged cells did not contain a free liquid phase. The remaining liquid (6 ml) was placed in the test tube, and two specimens inserted. One coiled specimen was suspended by a glass hook totally immersed, and one straight specimen was suspended so that it included the interface. The tube was covered by the film, held in place by wire, and the tubes immersed in the water bath. After the end of one week exposure, the specimens were removed, cleaned in room temperature hydrochloric acid inhibited by stannous and antimony chlorides, rinsed, dried, and reweighed.

- (c) Reactive waste: Three test procedures were used to determine reactivity:

—The top of the cell was removed, and the cell immersed in approximately 2 liters of water. Gases evolved were sampled by syringe and analyzed chromatographically.

—The top of the cell was removed, and the cell immersed in 2 liters of 0.1N acetic acid. The gases evolved were absorbed in a 1.25N sodium hydroxide solution and analyzed for cyanide using the colorimetric method described in "Methods for Chemical Analysis of Water and Wastes," EPA 600/4-79-020, March 1979.

—The cells were placed in an oven at 204°C (400°F) and observed.

- (d) Toxic waste: The procedures given in the proposed rules, Section 250.13(d)(2), were used. The filtration method for separation was used. The anode, cathode, and separator were cut into pieces smaller than 9.5 mm (0.375 in) before the extraction procedure. Stirring motor speed was 60 rpm. The pH was controlled manually. Analysis was accomplished with a Perkin-Elmer Model 503 Atomic Adsorption Spectrophotometer with an HGA 2100 graphite furnace. Test procedures for analysis are those described in "Methods for Chemical Analysis of Waters and Wastes," EPA 600/4-79-020, March 1979.

b. Test Results

The determination of the hazardousness of the waste based on the use of representative samples of the contents of the cells is presented below according to the criteria presented in the various paragraphs of Section 250.13 of the proposed rules.

- (a) Ignitable waste:

(a)(1)(i): The criteria from this paragraph read as follows:

. . . is a liquid and has a flash point less than 60°C (140°F) determined by the method cited below or an equivalent method.

These criteria were not applicable to the discharged cells from either manufacturer, as there was no free liquid in the cells. Free liquid was obtained from the fresh cells manufactured by both Mallory and PCI when the cells were opened at -40°C (-40°F) as described earlier in Section 2, Waste Sampling. When the liquid from either cell was tested using the Setaflash Closed Tester, at 60°C (140°F), there was no flash; in fact, the pilot flame was extinguished, apparently due to sulfur dioxide coming from the cup. Thus, a representative sample of the liquid portion of the waste did not flash at the test temperature, and the waste is not considered hazardous according to these criteria.

(a)(1)(ii): The criteria from this paragraph read as follows:

. . . is not a liquid and is liable to cause fires through friction, absorption of moisture, spontaneous chemical changes, or retained heat from manufacturing or processing, or when ignited burns so vigorously and persistently as to create a hazard during its management.

The criterion of concern in this paragraph is the question of burning in a vigorous or persistent manner when ignited. As discussed previously, the cells are encased in a strong steel case, but can be ruptured with the application of sufficient force. To simulate this case, the tops of discharged and fresh cells of both Mallory and PCI manufacture were removed, and it was attempted to ignite the open cells with a propane torch. When flame was played on the open cell, the contents burned with the characteristic red flame of lithium, but the flame was not vigorous, and the flame extinguished shortly after the torch was removed. Thus, while the cells were ignitable, they did not meet the criteria for hazardoussness.

If the contents of the cells were removed, the spiral unwound, and the separate components ignited, the lithium from the fresh cells burned in a manner best described as rapid but not vigorous, but of such a persistent manner that it could be difficult to control. However, because this unwound condition is not one that would be encountered in disposal practice, it was not considered as indicating a hazardous waste.

(a)(1)(iii) and (iv): The criteria from these paragraphs read as follows:

. . . is an ignitable compressed gas as defined in 49 CFR 173.300(b), or is an oxidizer as defined in 49 CFR 173.151.

Neither of these criteria apply to any portion of this waste.

• (b) Corrosive waste:

(b)(1)(i) and (ii): These criteria read as follows:

. . . is aqueous and has a pH less than or equal to 3 or greater than or equal to 12 as determined by the method cited below or an equivalent method, or corrodes steel (SAE1020) at a rate greater than 0.250 inch per year at a test temperature of 130°F as determined by the method cited below or an equivalent method.

The criteria do not apply to the discharged cells because there was no liquid present in these cells. The pH criteria do not apply to the fresh cells, as they are not aqueous. Most of the liquid present in the fresh cells evaporates at the specified test

temperature of 130°F, leaving only a small amount of liquid, which is probably primarily acetonitrile. When tested for corrosiveness toward steel, no corrosion could be detected, so that the waste was determined to be non-corrosive according to the proposed rules.

• (c) Reactive waste:

(c)(1)(i): The criteria from this paragraph read as follows:

. . . is normally unstable and readily undergoes violent chemical change without detonating; reacts violently with water, forms potentially explosive mixtures with water, or generates toxic gases, vapors, or fumes when mixed with water; or is a cyanide or sulfide bearing waste which can generate toxic gases, vapors, or fumes when exposed to mildly acidic or basic conditions..

The components of the cells were investigated for hazards as listed in these criteria. The cells contain lithium, which is known to react rapidly with water, generating heat and hydrogen. To determine the reaction between a ruptured cell and water, the top was removed from each type of cell, and it was placed in water. In all cases there was an evolution of gases when the cells were placed in water. The reaction was more rapid for the fresh cells than for the discharged cells, but in no case could the reaction be described as "violent." It appears that the tight spiral configuration of the internals of the cells slows diffusion of water to the lithium, and prevents violent reaction.

The gases released when the cells were allowed to react with water were analyzed. The gas was primarily hydrogen, with small amounts of sulfur dioxide (0.5% to 1.6%). The cells were exposed to mildly acidic conditions (0.1N acetic acid), and the evolved gases were analyzed for cyanide. None was detected; the limit of detectability was estimated at 0.1 mg HCN.

One of the criteria in this paragraph is met to some degree: ". . . or generates toxic gases, vapors, or fumes when mixed with water." Some sulfur dioxide is contained in the gases evolved by reaction with water, but the amount generated per cell is small (0.10 to 0.31 grams).

(c)(1)(ii): The criteria from this paragraph read as follows:

. . . is capable of detonation or explosive reaction but requires a strong initiating source or which must be heated under confinement before initiation can take place, or which reacts explosively with water.

As discussed in the section dealing with tests of the whole cells, there is no explosive reaction when heated under confinement.

(c)(1)(iii) and (iv): The criteria from these paragraphs read as follows:

. . . is readily capable of detonation or of explosive decomposition or reaction at normal temperatures and pressures, or is a forbidden explosive as defined in 49 CFR 173.51, a Class A explosive as defined in 49 CFR 173.53, or a Class B explosive as defined in 49 CFR 173.58.

The components of the cell are not capable of detonation or explosive reaction at normal temperatures and pressures, are not listed explosives and are not hazardous under these criteria.

• (d) Toxic waste:

(d)(1): The criteria in this section deal with the determination of the toxicity of a waste by analysis of materials leached from the waste under specified conditions. The materials specified for analysis are 8 metals and 6 pesticides. The pesticides are not of concern in the cells, but the leaching and analysis procedure was carried out for the 8 metals. The results of tests using the internals from both fresh and discharged cells from both Mallory and PCI are given in Table 2. It can be seen that none of the cells produced a leachate containing concentrations of the metals above that specified in the proposed rules, and thus are not considered hazardous under these criteria.

Table 2. Extraction Procedure Results.

		Extract Level, mg/l					Contamination Limit
Cell Mft.:		PCI		Mallory			
State:		Discharged	Fresh	Fresh	Discharged	Fresh	
Elements	As	<0.050	<0.050	<0.050	<0.050	<0.050	0.50
	Cd	0.035	0.031	0.035	0.029	0.066	0.10
	Cr	0.016	0.007	0.004	0.003	0.004	0.50
	Pb	<0.500	<0.500	<0.500	<0.500	<0.500	0.50
	Ba	0.124	0.023	0.071	0.096	0.071	10.00
	Se	<0.100	<0.100	<0.100	<0.100	<0.100	0.10
	Hg	<0.001	<0.001	<0.001	<0.001	<0.001	0.02
	Ag	0.160	0.006	0.110	0.140	0.060	0.50

Note: State = either discharged or fresh.
Duplicate tests were run on fresh Mallory cells to determine repeatability.

The following information is given here as being of interest, but having no direct effect on determination of hazardousness. In all cases the internals from 3 cells were used for the test, having sample weights ranging from 101 to 105 grams. Both of the fresh cell samples (Mallory and PCI) used the entire 4 ml of acid solution per gram of sample without reaching 4.8 to 5.2 pH range. The final pH values after 24 hours of leaching were 7.8 for the Mallory cell and 8.0 for the PCI cells. The discharged PCI cell also required the entire 4 ml of acid solution per gram, and reached a final pH of 5.3. The discharged Mallory cell required a total of 2.3 ml of acid solution per gram to reach a final pH in the desired range (5.2).

c. Instruments Used in Test Procedures

- Setaflash Closed Cup Tester, Stanhope-Seal, Ltd., Model 1374
- Gas Chromatograph, Varian Model 3700
- Perkin Elmer Model 503 Atomic Adsorption Spectrophotometer, with HGA 2100 Graphite Furnace
- pH Meter, VWR Scientific Cat. No. 34100-006.

d. Anticipated Changes in Waste

The cells manufactured by both Mallory and Power Conversion, Inc., have been tested. It is believed that these cells represent the present and near-future cells which will be used. Advances in technology are not expected to significantly change the components of the cells. Retesting should not be required unless significant changes in components or structure are made.

3. Summary of Test Results

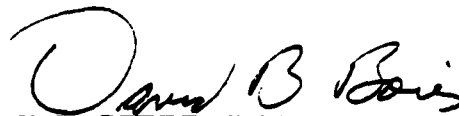
Application of the tests and criteria in Section 250.13 of the proposed rules show a certain amount of reactivity of the cells towards water if the case is ruptured, but not of a degree to be considered hazardous. The tests and criteria of the proposed rules show a small release (0.1 to 0.3 grams/cell) of a toxic gas, sulfur dioxide when water contacts the cell when the case is ruptured. In actual practice, if the cells were ruptured, a much larger amount of sulfur dioxide would be released as the internal pressure was vented, although this behavior is not covered by the proposed rules.

In summary, considering the results of both the individual cell tests and the tests using the cell internals, the following conclusions can be drawn:

- The cells are not hazardous for disposal if the case is not ruptured.
- On the basis of the Structural Integrity Procedure of the proposed rules, the cells will not rupture and, thus, will not be considered hazardous.
- If the cell case should be ruptured in some manner, a small amount (approximately 2 to 12 grams) of sulfur dioxide will be released. It is an irritant, odorous, and toxic gas. The degree of hazard will depend on the number of cells ruptured at a time, meteorological conditions, the distance to sensitive receptors, etc.

D. CERTIFICATION

I have personally examined and am familiar with the information submitted in this certification, and I hereby certify under penalty of law that this information is true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment.



David B. Boies
Senior Chemical Engineer

APPENDIX

Section 250.13, Proposed Amendment of 40 CFR, Part 250
Federal Register, Vol. 43, No. 243
December 18, 1978

§ 250.13 Hazardous waste characteristics.

(a) **Ignitable waste.** (1) **Definition**—A solid waste is a hazardous waste if a representative sample of the waste:

(i) Is a liquid and has a flash point less than 60°C (140°F) determined by the method cited below or an equivalent method, or

(ii) Is not a liquid and is liable to cause fires through friction, absorption of moisture, spontaneous chemical changes, or retained heat from manufacturing or processing, or when ignited burns so vigorously and persistently as to create a hazard during its management, or

(iii) Is an ignitable compressed gas as defined in 49 CFR 173.300(b), or

(iv) Is an oxidizer as defined in 49 CFR 173.151.

(2) **Identification method.** (i) Flash point of liquids shall be determined by a Pensky-Martens Closed Cup Tester, using the protocol specified in ASTM Standard D-93-72, or the Setaflash Closed Tester using the protocol specified in ASTM standard D-3278-73 or any other equivalent method as defined in this Subpart.

(ii) Ignitable gases shall be determined by the methods described in 49 CFR 173.300.

(b) **Corrosive waste.** (1) **Definition**—A solid waste is a hazardous waste if a representative sample of the waste:

(i) Is aqueous and has a pH less than or equal to 3 or greater than or equal to 12 as determined by the method cited below or an equivalent method, or

(ii) Corrodes steel (SAE 1020) at a rate greater than 0.250 inch per year at a test temperature of 130°F as determined by the method cited below or an equivalent method.

(2) **Identification method.** (1) pH shall be determined using a pH meter, following the protocol specified in the "Manual of Methods for Chemical Analysis of Water and Wastes" (EPA-625-16-74 003).

(ii) Rate of metal corrosion shall be determined using the protocol specified in NACE (National Association of Corrosion Engineers) Standard TM-01-69.

(c) **Reactive waste.** (1) **Definition**—A solid waste is a hazardous waste if a representative sample of the waste:

(i) Is normally unstable and readily undergoes violent chemical change without detonating; reacts violently with water, forms potentially explosive mixtures with water, or generates toxic gases, vapors, or fumes when mixed with water; or is a cyanide or sulfide bearing waste which can generate toxic gases, vapors, or fumes when exposed to mild acidic or basic conditions.

(ii) Is capable of detonation or explosive reaction but requires a strong initiating source or which must be heated under confinement before initiation can take place, or which reacts explosively with water.

(iii) Is readily capable of detonation or of explosive decomposition or reaction at normal temperatures and pressures.

(iv) Is a forbidden explosive as defined in 49 CFR 173.51, a Class A explosive as defined in 49 CFR 173.53, or a Class B explosive as defined in 49 CFR 173.58.

NOTE—Such waste includes pyrophoric substances, explosives, autopolymerizable material and oxidizing agents. If it is not apparent whether a waste is a hazardous waste using this description, then the methods cited below or equivalent methods can be used to determine if the waste is hazardous waste.

(2) **Identification method.** (1) Thermally unstable waste can be identified using the Explosion Temperature Test cited in Appendix II of this Subpart (waste for which explosion, ignition, or decomposition occurs at 125°C after 5 minutes is classed as hazardous waste).

(ii) Waste unstable to mechanical shock can be identified using the Bureau of Explosives impact apparatus and the tests cited in 49 CFR 173.53(b), (c), (d), or (f), as appropriate.

(d) **Toxic waste.** (1) **Definition**—A solid waste is a hazardous waste if, according to the methods specified in paragraph (2), the extract obtained from applying the Extraction Procedure (EP) cited below to a representative sample of the waste has concentrations of a contaminant that exceeds any of the following values:

Contaminant	Extract level, milligrams per liter
Arsenic	0.50
Barium	10.0
Cadmium	0.10
Chromium	0.50
Copper	0.50
Mercury	0.02
Selenium	0.10
Silver	0.50
Endrin (1,2,3,4,10,10-hexachloro-5, 7-epoxy-1,4,4a,5,6,7,8,8a-octahydro-1, 4-endo, endo-5, 8-di methano naphthalene)	0.002
Lindane (1,2,3,4,5,6-hexachlorocyclohexane gamma isomer)	0.040
Methoxychlor (1,1,1-Trichloroethane 2,2-bis (p-methoxyphenyl))	1.0
Toxaphene (C ₁₂ H ₈ Cl ₆ -technical chlorinated camphene, 87-89 percent chlorine)	0.050
2,4-D (2,4-Dichlorophenoxyacetic acid)	1.0
2,4,5-TP Silver (2,4,5-Trichlorophenoxypropionic acid)	0.10

NOTE—Extract levels specified for the above substances equal ten times the EPA National Interim Primary Drinking Water Standards for these substances. These standards are being revised. Extract levels specified above will be changed to reflect revisions to these standards. Also, EPA is considering use of the Water Quality Criteria under the Clean Water Act as a basis for setting extract levels. In addition to the EPA National Interim Primary Drinking Water Standards.

(2) **Identification method.** (i) **Extraction procedure.** (A) Take a representative sample (minimum size 100 gms) of the waste to be tested and separate it into its component phases using either the filtration method or the centrifugation method described in this section. Reserve the liquid fraction under refrigeration at 1-5°C (34-41°F) for use as described in paragraph (F) of this section.

(1) Filtration Method.

Equipment: Millipore YY22 142 30 filter holder (Millipore Corp., Bedford, MA 01730) equipped with an X142 142 08 accessory 1.5 liter reservoir, or Nuclepore Corp., Pleasanton, CA 94566) equipped with a 1.5 liter reservoir, or equivalent filter holder.

Procedure: 1. Using the filter holder place a 0.45 micron filter membrane (Millipore type HAWP142, Nuclepore type 112007, or equivalent) on the support screen. On top of the membrane (upstream) place a prefilter (Millipore AP25124, Nuclepore P040, or equivalent). Secure filter holder as directed in manufacturer's instructions.

2. Fill the reservoir with the sample to be separated, pressurize to no more than 75 psi (7 kg/cm²), and filter until no significant amount of fluid (0.5 ml) is released during a 30 minute period.

3. After liquid flow stops, depressurize and open the top of the reservoir, invert the filter unit, replace filter pads as in step 1, above, and resume filtering. Save pads for later use. Repeat this step until no more fluid can be removed from the waste at a pressure of 75 psi (7 kg/cm²).

4. Take the solid material, and any pads used in filtration, and extract as described in paragraph (B). Subtract tare weights of filter pads in calculating the amount of solid material.

(2) Centrifugation Method

Equipment: Centrifuge (e.g. Damon-IEC catalog no. 7165, Damon-IEC Corp., Needham Heights, MA. or equivalent) equipped with a rotor for 500 ml to 1 liter containers (Damon-IEC catalog no. 978, or equivalent). For flammable material containing wastes, explosion proof equipment is recommended.

GLASS CENTRIFUGE BOTTLES

Procedure: 1. Centrifuge sample for 30 minutes at 3300 rpm. Hold temperature at 20-40°C (68-104°F).

2. Using a ruler, measure the size of the liquid and solid layers, to the nearest mm (0.40 inch). Calculate the liquid to solid ratio.

3. Repeat 1 and 2 above until the liquid: solid ratio calculated after two consecutive 30 minute centrifugations is within 3%.

4. Decant or siphon off the layers and extract the solid as described in paragraph B.

(B) Take the solid portion obtained in paragraph (1), and prepare it for extraction by either grinding it to pass through a 9.5 mm (3/8") standard sieve or by subjecting it to the following structural integrity procedure.

STRUCTURAL INTEGRITY PROCEDURE

Equipment: Compaction tester having a 1.25 inch diameter hammer weighing 0.73 lbs. and having a free fall of 3 inches (Figure 1) (one suitable device is the Associated Design and Manufacturing Company, Alexandria, Va. 22314, catalog no. 125).

Procedure: 1. Fill the sample holder with the material to be tested. If the waste sample is a monolithic block, then cut out a representative sample from the block having the dimensions of a 1.5" dia. x 2.5" cylinder.

2. Place the sample holder into the Compaction Tester and apply 15 hammer blows to the sample.

3. Remove the now compacted sample from the sample holder and transfer it to the extraction apparatus for extraction.

(C) Take the solid material from paragraph (B), weigh it and place it in an extractor. A suitable extractor will not only prevent stratification of sample and extraction fluid but also insure that all sample surfaces are continuously brought into contact with well mixed extraction fluid. (When operated at greater than or equal to 40 rpm, one suitable device is shown in Figure 2 and available as Part No. 3736 produced by the Associated

Design and Mfg. Co., Alexandria, Va. 22314.)

(D) Add to the extractor a weight of deionized water equal to 15 times the weight of solid material added to the extractor. This includes any water used in transferring the solid material to the extractor.

(E) Begin agitation and adjust the pH of the solution to 5.0±0.2 using 0.5N acetic acid. Hold the pH at 5.0±0.2 and continue agitation for 24±0.5 hours. If more than 4 ml of acid for each gm of solid is required to hold the pH at 5, then once 4 ml of acid per gm has been added, complete the 24 hour extraction without adding any additional acid. Maintain the extractant at 20-40°C (68-104°F) during extraction. It is recommended that a device such as the Type 43-A pH Controller manufactured by Chemtrix, Inc., Hillsboro, OR 97123, or equivalent, be used for controlling pH. If such a device is not available then the following manual procedure can be employed.

MANUAL pH ADJUSTMENT

1. Calibrate pH meter in accordance with manufacturer's specifications.

2. Add 0.5N acetic acid and adjust pH of solution to 5.0±0.2. If more than 4 ml of acid for each gm of solid is required to hold the pH at 5, then once 4 ml of acid per gm has been added, complete the 24 hour extraction without adding any additional acid. Maintain the extractant at 20-40°C (68-104°F) during extraction.

3. Manually adjust pH of solution at 15, 30, and 60 minute intervals moving to the next longer interval if the pH did not have to be adjusted more than 0.5 pH units since the previous adjustment.

4. Continue adjustment procedure for a period of not less than 5 hours.

5. Final pH after a 24 hour period must be within the range 4.9-5.1; unless 4 ml of acid per gram of solid has already been added.

6. If the conditions of 3 are not met, continue pH adjustment at approximately one hour intervals for a period of not less than 4 hours.

(F) At the end of the 24 hour extraction period, separate the material in the extractor into solid and liquid phases as in paragraph (A). Adjust the volume of the resulting liquid phase with deionized water so that its volume is 20 times that occupied by a quantity of water at 4°C equal in weight to the initial quantity of solid material charged to the extractor (e.g., for an initial weight of 1 gm, dilute to 20 ml). Combine this solution with the original liquid phase from paragraph (A). This combined liquid, and any precipitate which may later form, is the Extraction Procedure Extract.

(II) Analysis—Analyses conducted to determine conformance with Section 250.13(b)(1) shall be made in accordance with the following or equivalent methods:

(A) Arsenic—Atomic Absorption Method, "Methods for Chemical Anal-

ysis of Water and Wastes," pp. 95-96, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(B) Barium—Atomic Absorption Method, "Standard Methods for the Examination of Water and Wastewater," latest edition, or "Methods for Chemical Analysis of Water and Wastes," pp. 97-98, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(C) Cadmium—Atomic Absorption Method, "Standard Methods for the Examination of Water and Wastewater," latest edition, or "Methods for Chemical Analysis of Water and Wastes," pp. 101-103, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(D) Chromium—Atomic Absorption Method, "Standard Methods for the Examination of Water and Wastewater," latest edition, or "Methods for Chemical Analysis of Water and Wastes," pp. 112-113, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(E) Lead—Atomic Absorption Method, "Standard Methods for the Examination of Water and Wastewater," latest edition, or "Methods for Chemical Analysis of Water and Wastes," pp. 112-113, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(F) Mercury—Flameless Atomic Absorption Method, "Methods for Chemical Analysis of Water and Wastes," pp. 118-126, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(G) Selenium—Atomic Absorption Method, "Methods for Chemical Analysis of Water and Wastes," p. 145, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(H) Silver—Atomic Absorption Method, "Standard Method for the Examination of Water and Wastewater," latest edition, or "Methods for Chemical Analysis of Water and Wastes," p. 146, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

APPENDIX I—SAMPLING METHODS

The methods and equipment used for sampling waste materials will vary with the form and consistency of the waste material to be sampled. Listed below are sampling protocols appropriate for sampling waste materials with consistencies similar to the indicated material.

Extremely viscous liquid—ASTM Standard D140-79

Crushed or powdered material—ASTM Standard D315-75

Soil or rock-like material—ASTM Standard D120-69

Soil-like material—ASTM Standard D1452-65

Fly ash-like material—ASTM Standard D2234-76

Additional protocols to be used are described in the draft report "Handbook for Sampling Hazardous Waste", Research Grant R-804692010, available from USEPA, Office of Solid Waste, Information-Materials Group, Cincinnati, Ohio 452168.

APPENDIX II—EXPLOSION TEMPERATURE TEST

1. *Purpose of Test:* To determine whether a material explodes, ignites, or decomposes after a five second immersion in a sand bath or low flammability liquid (such as high molecular weight silicone oil) at temperatures up to 125°C and if so, at what temperature.

2. *Operating Principle:* This test gives an estimate of how close the explosion temperature is to ambient condition for a material, and, hence, provides a measurable indication of thermal instability.

3. *Test Description:* The material to be tested (25 mg.) is placed in a copper test tube (high thermal conductivity) and immersed in the controlled temperature bath. This test is made at a series of bath temperatures, and the time lag prior to explosion at each temperature is recorded (up to 10 min.). The bath temperature is raised until a temperature of 125°C is reached if no explosion, ignition, or apparent decomposition occurs.

NOTE.—This is a modification of the test taken from H. Henkin, and R. G. McGill, Industrial & Engr. Chem. V44 p135.

Figure 2

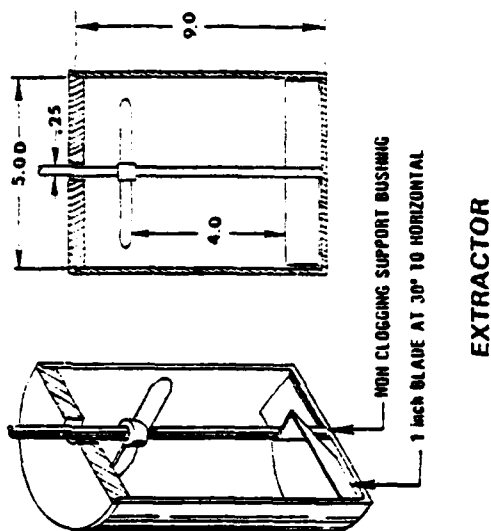
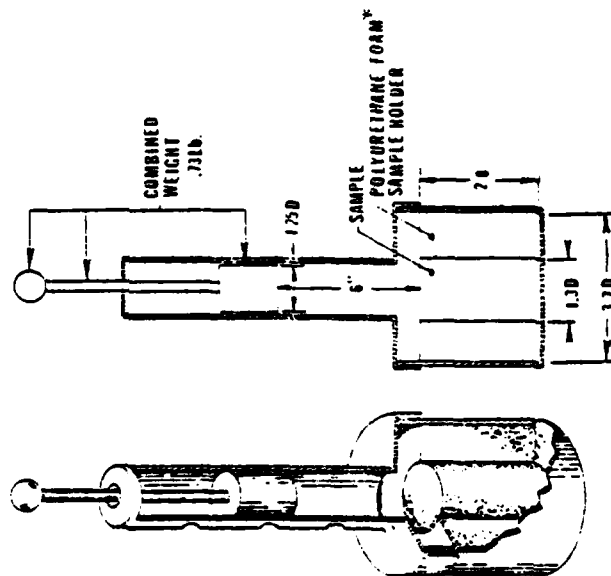


Figure 1



COMPACTION TESTER

* Polyurethane foam shall conform to requirements for Grade 21, performance Grade AD or BD, established in ASTM Standard D3453.

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